Notebook Series -- Notebooks by Experimenters Other Than Edison
Chemical Experiments -- L. F. Ott Books
Notebook N-18-09-12

This notebook was used by Ludwig F. (Louis) Ott during September 1918-April 1920 for notes on various chemical experiments. Most of the experiments pertain to chemical solutions used in making storage batteries or efforts to reuse elements from old storage batteries. Comments by Ott on some of the entries indicate Edison's attention to, and correction of, Ott's work. The front cover is marked "Note Book L. Ott." The book contains 192 numbered pages, some of which are blank.

7998 Z

Acme Co.,

MFG. STATIONERS,
95 JOHN ST.
AND
19 PLATT ST.
NEW YORK.

~~Big Dish 57.3590~~
~~Small Dish 29.3095~~
~~Crucible III 38.4685 38.4770~~ 38.4753
" ~~IV 44.5929~~

Small platinum dish 32.0790
Crucible # IV 44.5710

[illegible] 0.112
0.107
0.100

1107

Sept. 12, 18.

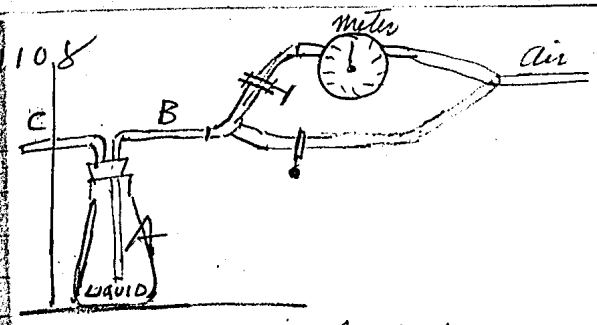
Experiment to make PbO_2 by electrical Deposition

Made an anode of PbS by melting Pb and adding Sulphur until the theoretical amount was taken up.
207 Pb require 32 Sulphur total weight 239 grams.
the anode was then cast by heating the mixture to a bright red and pouring into an iron mould, this did not cast very well as the mould was to cold this composition chills very readily. Anode was then sawed and filed smooth.

Wt of Anode + Copper Wire before placing in Cell = 25.8840 g

Sodium monosulphide solution Sp. G. 1100.
Current = .4 amp. 1 amp.
Volts
On 4.40 PM Sept. 12/18 on all night at 10 PM 13
" changed to $1\frac{1}{2}$ amp.

Anode covered with some free sulphur



Rough Test.

10cc or 19.34 grams of Oleum containing 60% free SO_3 was placed in bottle A which was connected by means of tube B to an air line, the fumes escaping through tube C which had an opening of 1/8"

Rate of air flow = about 54 cubic feet per hour

Started 5.20 finished 5.30 — 2 min. for belt coming off motor on air blower total time 8 minutes at the end of this time became weak of about 7 min. 19.34 x 40% = 7.736 grams should be left after all SO_3 had been applied but there was left. 8.234 grams. Quite a rise in temperature in bottle A

1109

Same as #1108 except used Antimony Penta Chloride 10 cubic centimeters

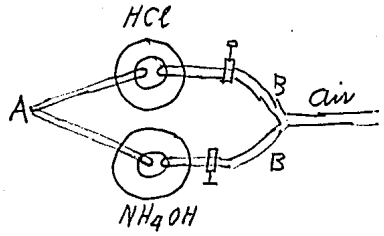
Off 9.59
On 9.52
7 minutes

Gives a white smoke about 80–90 % of that of the Oleum Smoke very heavy

1110

Same as #1108 except used Sulphur Chloride No smoke.

#1111



One bottle contained 10 cc HCl the other bottle contained 10 cc NH_4OH gas from both bottles meeting at A air was passed into the bottles from B.B. No smoke as the HCl did not evaporate fast enough

By Heating the bottle containing the HCl get smoke

Oct. 3, 1918

1112 Data Oct. 3 1918

H_2SO_4 + bottle 5020 grams before mixing
" + " 2560 " after "
2460 " or 5.4 lbs H_2SO_4 used.

Aniline 200 cc.

Container + Salt + Water = 7640 grams
H_2SO_4 consumed 2460 "
10,100 grams

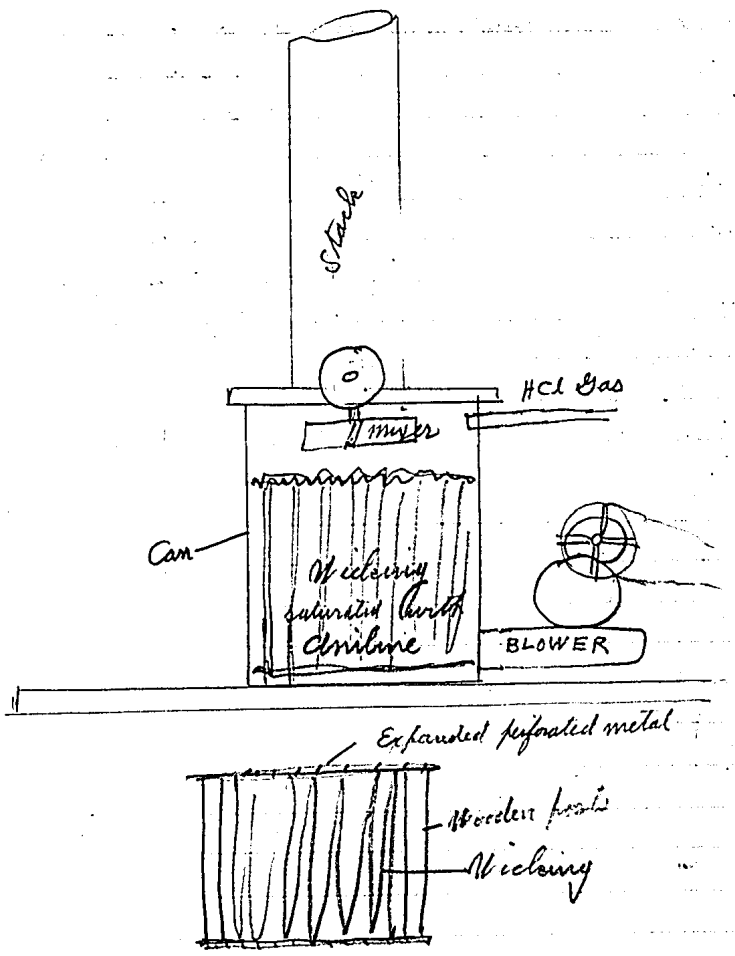
After run 10000
100 grams HCl gas

developed
Finish 3.40 start 2.20 = 1 hour 20 minutes
Box 9"x9"x13½
Space above wicks 9x9x5½
From top of wicks to center inlet hole for gas 2½"
Size of gas inlet tube 9/16"
Size of air inlet to blow gas out of generator 1/4"
Speed of fully on blower 23 rev per min.
Speed of fan blades
Size of blades
No of blades
Size of hole for air exhaust 2½" diam
Gear Ratio of driving shaft to blades 45¾ to 1
Speed of fan blades on Motor 780 rev per min
No of blades 4
Pitch of blades. X 20°

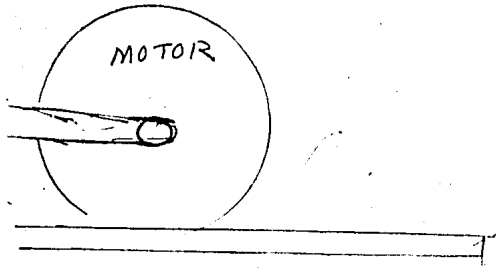
0

height of fan blade above mixer 2"
Size of Blade = 8" diam x 5 feet high.

200 cc aniline for 80 min = 2.5 cc aniline per minute
No of cubic feet of air from the blower = about 52 cubic feet per minute

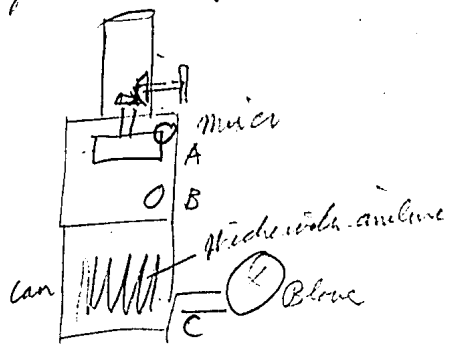


air from blower enters the can at the bottom of one side and passes over a mass of wicking strung back and forth on a frame which consisted of 2 pieces of expanded perforated metal, such as used to hold stucco on houses, held together by 4 wooden posts. Wicking saturated with aniline The air then passes over the meter where it is met by a stream of HCl gas entering the chamber from the side thence up the stack.



Oct 8, 18

Tried the following putting another can above the can in illustration and of the same size



Air entered at C passed over wicking containing aniline and went to the can above the HCl gas was tried at both entrances A and B and found very little difference this gave only about 50% of the smoke that the previous way gave showing that under these conditions it is necessary to have the HCl mix with the Aniline as near to the wicking as possible

By leading the HCl gas into the Blower it gives much more smoke but only for about 1/2 the time

as the Hydrochloride is formed on the wicking and diminishes the exposed surface of Aniline —

1113 Absorption of Ammonia by carbonized Condensite

20.1360	20.1396
18.1360	20.1360
2.0000 g material	.0036

2.) .0036.00 (18%
2/16

can 3.00 cor 2.2
off 9.00 cor 2.3 = 18 hrs
but found lid partly open.

Lost some in putting back
so reweighed 20.0780
20.0780
0 [illegible]

3 hrs

---

can 2.00

Wt. =	21.3800	21.3830
	18.1360	21.3800
	3.2440	.0030

absorbed in 18 hrs

Some condensite put in a crucible, covered with a lid having a hole in the center, thermometer inserted heated to 360° C using a nitrogen filled thermometer.
Kept at 360° for 5 minutes then placed in Dessicator to cool
When cool transferred to a weighing bottle with ground glass stopper and weighed

then put in an empty dessicator the lid removed, a rubber tube inserted, which had been previously connected to a cylinder of dry Ammonia Gas, Ammonia passed in for about 1 minute then the tube withdrawn and the lid of dessicator closed left 18 hours then taken out and weighed

This time the dessicator lid was still on tight or no ammonia escaped.

1114 L.O. Oct. 21/18

Making a crystalline Copper Hydroxide $Cu(OH)_2$

#1 25g $CuSO_4$ dissolved in 400 cc H_2O Prec Basic Salt by running in Ammonia with a burett until supernatant liquid just turned blue, Filtered washed, then converted into the Hydrate by covering with a 6% NaOH solution cold under vigorous stirring for about 10 minutes filtered washed and dried in Vacuum Room Temperature.

Remarks Not Crystalline

#2 25g $CuSO_4$ dissolved in 400 cc H_2O Prec. basic salt by letting in Ammonia (about 25 cc of the C.P. Ammonia) by means of a pipette letting the pipett down to the bottom of the beaker precip. boiling hot Washed Filtered then converted into the Hydrate by pouring on 6% NaOH cold under vigorous stirring Washed dried in Vacuum Room Temp.

Remarks Slightly Crystalline

See page 60 for #3A

#3 Rough Experiment. Used about a 30% Sol. $CuSO_4$ added ammonia until precip of basic salt was redissolved cold. then added Strong NaOH. 20% Washed by decantation filtered, dried in Air Room Temp.

Remarks Crystalline not first class. Try much different stands KOH

Duplicate Made 200 cc 30% $CuSO_4$ $5H_2O$ + 100 cc 28% Ammonia Poured 150 cc 20% NaOH into this without letting it cool washed by decantation filtered dried in Air Room Temp.

1114

#4 25g $CuSO_4$ in 400cc H_2O added ammonia by means of pipett while boiling hot (about 25cc) to form basic salt. Filtered washed then converted into Hydrate by pouring on 6% Solution KOH 400cc under vigorous stirring for about 10 min filtered washed dried in air. Room Temp.

Remarks a little Crystalline
No good ~~[illegible]~~ blade in KOH

#5 Same as #2 but dried 7cm inches above hot plate.

#6 Same as #2 but dried 10 inches above hot plate

#7 25 Grams $CuSO_4$ in 400cc H_2O added ammonia by means of pipett while boiling hot (about 25cc) to form basic salt. Filtered, washed then converted into Hydrate by pouring on 6% Solution LiOH 400cc under vigorous stirring for about 10 minutes filtered washed dried in air Room Temperature

Crystalline but not good

#8 Some Cupric Ammonium Sulfate from Powers Weightman which is in good crystalline form put into a beaker and 400cc of 6% NaOH poured over, stirred vigorously for 5 min then washed by decantation and filtered dried in air.
Settles very rapidly.

(1500)

#9 100 cc Copper Sulphate Solution Sp. G 1135 containing 20 g $CuSO_4 5H_2O$ per 100 cc or
" 12.75 g $CuSO_4$ " " "
To this 100 cc 1135 $CuSO_4$ solution was added (375) 25 cc of 0.900 Sp. G. Ammonia = to 28% NH_3
Temperature rose from 25° C to 40° C
Cooled in ice box to 12° C then poured into (1500) 100 cc of 20% NaOH also cooled to 12° C under constant stirring
Let stand 15 min. Then washed by decantation filtered and dried in air.
Note by mixing the temp dropped to 9° C
When first diluting the Cu solution the NaOH dissolves the Cu.
5 hours at 117° F Brown on bottom

10 100 cc 20% $CuSO_4 5H_2O$ Solt 25 cc 28% Ammonia cooled to room temperature then poured into 100 cc 20% NaOH Sol, also room temp, 30° C dropped to 25° C in mixing.
Same remarks as with #9
But it can be molded #9 #7 settles down much more than #10
Lasting about 1/2" light as against 1" both masses of same shape
3 hours at 117° F Brown on bottom

11 100 cc 20% $CuSO_4$ Solution added 25 cc Ammonia cooled to 12° C poured into 100 cc 20% NaOH [illegible] equal to a 10% [illegible] of [illegible] 12° C under constant stirring washed by decantation filtered and dried in air [illegible]
3 hours at 117° F Brown on bottom

When dried in a heat does not fuse immediately.

12 100 cc 20% $CuSO_4$ Solution added 25 cc Ammonia cooled to [illegible] poured into 100 cc 20% [illegible] NaOH Sol [illegible] room temp. washed by decantation dried in air
Room temp
2 1/2 hours at 117° F brown on bottom

When poured in a precip forms immediately

13 100 cc of 20% $CuSO_4$ sol to which was added 25 cc of 28% Ammonia poured into 100 cc 20% NaOH diluted with 300 cc H_2O = to a 5% NaOH sol.
Both warmed to 12° C under constant stirring washed by decantation followed dried on air.

Cuprous oxide also a small quantity is added.
2½ hours at 117 Brown on bottom

14 100 cc of 20% $CuSO_4$ sol to which was added 25 cc of 28% Ammonia poured into 100 cc of 20% NaOH diluted with 300 cc H_2O at room temp. Washed by decantation followed dried in air Room temp.

4½ hours at 117°F Brown on bottom

15 4000 cc of an 8% solution of Sodium Nitrate Copper Anode Graphite Cathode
Current density 2.5 amp. Volts = 5 V.
Time of Run 10.00 to 3.00 = 5 hours.
Some $Cu(OH)_2$ also settle on bottom anode scales of metallic copper which seem to have peeled off. Amperage approximate 232 CP and 114 CP.
11 hours at 120 Brown on bottom.

No of Grams = 17
$\frac{17}{5}$ = 3.54 grams per hour
2.5 X 5 12.5 Watts per hour
$\frac{3.54}{12.5}$ = .2832 grams per watt hour

or 283.2 grams per Kilowatt hour
$\frac{283.2}{453}$ = .625 pound per Kilowatt hour

1/14 25

#16 Rough experiment $Cu(OH)_2$ made by Naugora dissolved in Ammonia left to crystallize mother liquor drained off and crystals dried in air then poured 5% NaOH over the crystals under constant stirring room temp. washed by decantation filtered dried in air. Room temp.

1½ hours at 120°F. Brown on bottom

These samples were tested as follows, 250 milligrams of the hydrate were put on a watch glass in a glass the watch glass being raised 1 inch from the bottom of the glass by means of an iron wire frame filled up with 20% NaOH to 1¼ inches above the watch glass then put in oven at constant temperature. observations taken every ½ hour.

Exp. # 1115

Distillation of Benzene

cc	°Cent	°F	°F Vacuum of 24 1/2 to 25"	28" Vacuum °F
0	142	287	210	186
2	159	318	220	194
5	165	329	240	204
10	173	343	254	212
20	184	363	266	226
30	188	370	280	212
40	195	383	290	254
50	203	397	306	264
60	212	414	324	278
70	220	428	340	292
80	230	446	378	310
90	249	480	388	340
95	260	500	400	368

92 cc Limit of Thermometer

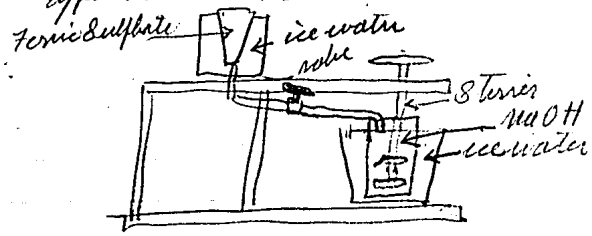
1114 # 17 $CuSO_4$ precip by pouring into NaOH containing a little glycerine $Cu(OH)_2$ dissolves to a blue solution, then precip $Cu(OH)_2$ by neutralizing with HCl, filtering, washing and drying in air room temp.

18 Put some of the soluble $Cu(OH)_2$ sol on a watch glass 4" above hot plate crystallized in needles but these were soluble in water —

19 50 cc 20% $CuSO_4$ sol added 12 cc 28% NH_4OH and run this into 50 cc 20% NaOH diluted with 50 cc H_2O which was put into a distilling flask with a vacuum of 24–28" mercury. temp of boiling = 120–125°F.

Turned dark

20 166 grams Ferrous Sulphate dissolved in 1000 cc Water added HNO_3 while boiling until all oxidized to Ferric Sulphate (Ferrocyanide of Potash Test) cooled in ice box. Then poured into a solution of 400 cc 20% NaOH diluted with 1600 cc H_2O adding it drop by drop under constant stirring kept to 40°F by ice water in a stirring apparatus constructed thus.



Washed by decantation room temp until free of Sulphates ($BaCl_2$ Test).

Filtered and dried in air.

Exp. #1114 #21

160 g $CuSO_4$ dissolved in 2000 cc H_2O
added 2000 cc Sodium Hypochlorite Solution
until there was no more precip. let stand
hours then washed by decantation until
free of Sulfates ($BaCl_2$ Test) filtered
and dried in air.

Note there was no more precipitate so added
350 cc more Hypochlorite, did not turn black.
so added about 100 cc 20% NaOH
turned black instantly.

Exp # 1114 #22

50 grams Chromic Nitrate were dissolved
by heating in 200 cc Water then cooled to room
temperature this was then poured slowly with
300 cc 20% NaOH all dissolved to a clear
green liquid, then boiled for 20 minutes
precipitating green Chromium Hydrate
washed by decantation until free of alkali and
nitrates filtered and dried in air overnight.

Nov, 18, 18.

Exp. # 1116
20% Caustic Soda in Battery Jar covered with $\frac{3}{16}$" layer of Stock Oil On Nov. 18, at 11.00 A.M. marked two ways Top of Oil Layer and Junction of the NaOH Solution and Stock Oil.

Nov. 18, 18.

Exp. (Test) 1117
Determination of B.P. at $27\frac{1}{2}$ mm (Hg) Vacuum Naroline NOS Stand. Oil Co. and Texaco Spirits from Texas Oil Co.

Naroline NOS		Texaco Spirits	
cc	F°	cc	F°
0	168	0	150
2	172	2	160
5	174	5	168
10	176	10	174
20	178	20	182
30	181	30	186
40	186	40	187
50	190	50	190
60	196	60	194
70	202	70	202
80	208	80	210
90	220	90	230
95	232	95	242

Nov. 18/18

Exp. #1118 Final Method to Recover Lithium from Used Battery Sol.

Rough Experiment

100 cc of Solution taken passed CO_2 precipitate formed filtered off washed a little with water dried Weight. = 1.020 weighed as Li_2CO_3 = .661 g LiOH

100 cc Solution Analysis as follows 18.07 g/l NaOH + 15.18 g/l LiOH per liter passed CO_2 gets very warm, precipitate, filtered off washed the precip. with a little water, then passed CO_2 thru the filtrate for 2 hours, no more precip. Filtrate then made nearly acid with HCl boiled and precip Lithium with Sodium Phosphate Na_2HPO_4 filtered and washed with Water 1/2 Ammonia 1/2. dried at 100°C and weighed 15.18 g LiOH = 28.0 Carbonate

CO_2 precip = 0.375 carbonate
Left in Solution = 1.420 carbonate
1.795

= 15.

2x 7.03

Rough Experiment not weighed totally accurate 100 cc of Sodium Lithium Removal Solution brought to a boil added saturated solution of Sodium Phosphate filtered with hot washed with 1/2 Ammonia 1/2 Water dried at 100°C weighed
found 2.1080 grams Li_3PO_4

2.1080 X factor .1817 = .383 g Li

$\frac{24 \times .383}{7}$ = 1.313 g LiOH

No 1 100 cc Sodium Lith Removal Solution containing 15.18 g LiOH per liter added 400 cc Water made just acid with H_2SO_4 then just alkaline with NaOH Boiled precip Lith with Sodium Phosphate conc, Solution filtered hot washed with Water until free of Sulphate dried at 100°C weighed 14.4100
12.5764
1.8336 Li_3PO_4 X .1817
= .333 grams Li $\frac{333 \times 24}{7}$ 1.141 g LiOH

2 100 cc Sodium Lithium Removal Solution diluted with 200 cc Water precip. $LiOH$ with H_3PO_4 filtered warm washed with water until free of alkali Litmus Paper Test, dried in Oven at 100°C. weighed

found 2.3270 grams Li_3PO_4

$2.3270 \times .1817 = .4228$ Li

or $\frac{.4228 \times 24}{7} = 1.449$ g $LiOH$

about 95%

Rough Experiment

Lithium Fluoride heated in a platinum dish with Sulfuric Acid until fumes of H_2SO_4 and no more HFl (Glass test) came off then diluted with water SO_4 precip with $Ba(OH)_2$ according to the following equation

$$2\,LiFl + H_2SO_4 = Li_2SO_4 + 2\,HF$$

$2 \times 26 = 52$g 98g 110g

$$Li_2SO_4 + Ba(OH)_2 = 2\,LiOH + BaSO_4$$

110g 315g

Found that the LiOH was free of acidified with HCl and adding $BaCl_2$ gave no precipitate showing that there were no sulfates left.

Added to the LiOH some H_2SO_4 & got precip showing that a little $Ba(OH)_2$ had been added in excess.

Lithium Carbonate

100 g Renewal Solution evaporated to 1/2 its volume then passed CO_2 thru until the heating reaction was over dried on hot plate to dryness, then added a little water, filtered on vacuum filter and washed by adding a little water at a time until it seemed free of soda dried and weighed.

Found 1.05 g carbonate should be 2.34 g. so passed CO_2 thru filtrate for 1 hour no precip then acidified with HCl made slightly alkaline with NaOH, added Sodium Phosphate and got big precip of Lithium Phosphate.

Exp. 1119

Determination of Porosity of Regular CuO Plate from Primary Battery

A piece of CuO Plate weighing 125.630 grams was placed in a beaker with added by letting the water run down the side of the beaker until the plate was only 1/2 covered allowed to stand over night, taken out surplus water wiped off with a blotter and weighed

Wt. after immersion = 131.120
Wt before " = 125.630
5.49 Water absorbed

$\frac{5.49}{125.63} = .0437$ g water absorbed per gram material.

Exp. #1114 # 23
Made the same as #9 except let the precip. stand 15 hours before washing (Room Temp.) by decantation.

24 Made same as # 9 except let precip stand 24 hours in ice box before washing by decantation

25 Made same as #9 except used 36 grams Copper Ammonium Sulphate dissolved in 100 cc water & no ammonia added. Copper Ammonium Sulphate made by dissolving molecular proportions of Copper Sulphate and Ammonium Sulphate and crystallizing.

#3 Lithium From Amblygonite

10 g Amblygonite added enough water to make a paste then added 2 cc conc H_2SO_4 5 cc Water mixed thoroughly then evaporated to dryness and heated on the asbestos with strong Bunsen burner, cooled, leached out with boiling water, filtered, prec. Al, Ca, Fe with Na_2CO_3 filtered then divided into two portions. 1/2 evaporated to about 20 cc added Na_2CO_3 and brought to dryness then dissolved in least possible amount of water filtered and washed precip with a little water. The other 1/2 made slightly alkaline with 2 1/2 cc 20% NaOH then precip boiling hot Lithium with Na Phosphate. evaporated to dryness, added a little water and an equal amount strong ammonia washed with 1/2 water and 1/2 Ammonia, filtered and dried at 100 then heated in crucible and weighed.

Prec. as Carbonate = .230 = .0432 Li.

Prec as Phosphate = 1.8690 = .3352 Li

Total = 13.5045
Crucible = 12.5700
.9345
2
1.8690

= 3.35% Li.

#4 done the same as #3 except ~~no~~ 5cc water water and 10cc H_2SO_4 (great excess) excess driven off by heating.

Prec. as Carbonate = .305 = .057 L1

Prec. as phosphate = 2.66 = .478 L1

13.9000
12.5700
1.3300 x2 = 2.66 = 4.78% L1

All the wash water and mother liquor from the carbonate precip. gathered together made acid with HCl boiled to expell CO_2 then made slightly alkaline with NaOH and precc as phosphate

Total phosphate precip = = L1

#1 10g Amblygonite fused with 2g Sodium Bisulphate 5cc water kept at red heat 1/2 hour then ground in mortar and leached out hot water filtered, prec Al, Cu, Fe, with Na_2CO_3 filtered, divided in 2 portions
1/2 precipitated as carbonate same as #3 and 4 and the other 1/2 precip. as phosphate same as #3 and #4.

Prec as carbonate = .050 = .0094 L1

Prec as phosphate = .7280 = .1308 L1

Total = 12.9340
12.5700
.3640
2
.7280 = 1.3%

#2 same as #1 except used 5 grams Sodium Bisulphate

Prec as Carbonate = .350 = .066 L1

Prec. as phosphate = 1.3870 = .249 L1

Total 13.2635
12.5700
.6935
2
1.3870 = 2.49% L1

Lithium from beets.

3 lots of 100cc of Sodium Lith. Rincoral solution neutralized with HCl evaporated to dryness and extracted with

1st Alcohol Ether.

2nd Alcohol (Special Denatured)

3rd Acetone (commercial)

Alcohol Ether extr. done using 100cc of 1/2 alcohol 1/2 ether used this three times then mixed with 200cc 1/2 alcohol 1/2 ether and evaporated off the alcohol ether dried at 100 and weighed

#5 10 grams Amblygonite fused with
10 grams Sodium Bisulfate

#6 10 grams Amblygonite fused with 15 g
Sodium Sulfate

7 10 grams Ambigonite fused with 20 grams Sodium Bisulphate

8 1 gram Ambigonite treated with 10 cc HFC 2 cc H_2SO_4.

9 .5 grams amblygonite fused with 10 grams caustic soda

10 grams Lithium Phosphate in 50 cc H_2O passed CO_2 for 4 hours. room temp. Filtered washed dried then tested for CO_2

Total = 72.6410

1 g Amblygonite Smith Process

57.8400
57.4929
.3471 Chlorides

Gooch Method = 57.8400
Dish = 57.4929
Li_2SO_4 .3471 = 4.38 Li
=

Total = 38.5435
Crucible = 38.5055
K & Na Sulfates .0380 = 3.80 K+Na
Sulfates

Amblygonite NaOH Process 1g sample

Total	29.6960
Wt small dish	29.3750
Li_2SO_4	.3210
	.1262
	6420
	19260
	6420
	3210
	04.051020 = 4.05% Li

Received 50 cc of Solution to precipitate according to Scott page 346 to get rid of. Phosphoric Acid Iron Alumina Silica, Calc, Ba, Magnesia.

50 cc + 10 cc Wash water required .4 cc of 5% $BaCl_2$ Solution, then diluted with 100 cc water and boiled, required 3.8 cc of 26.75% NH_3 solution filtered half off and added 5 cc of a 15% Ammonium Carbonate solution and boiled the precipitate so filtered the other half off adding the two filtrates and washing the Precipitate until no more chlorides Silver Nitrate test, evaporated down to dryness and drove off the ammonia chloride at lowest possible heat. dissolved in water and brought up to 25 cc

#3A Exp. # 1114

Made same as #3 except that the supernatant liquid from the precipitate were poured off and allowed to evaporate the NH_3 in the air as the NH_3 solvent for $Cu(OH)_2$ evaporated off crystalline $Cu(OH)_2$ was formed. this is #3A.

Knight is trying variations —

Tested this 3.A for Sulfates by dissolving in HCl adding Barium Chloride and heating only a trace of Sulfates

Tested 1g by placing in a Kjeldahl Flask dissolving with a little H_2SO_4 then adding NaOH and boiling condensing the vapors and passing them through $\frac{1}{10}$ Normal H_2SO_4 and titrating back found just a trace 10 cc $\frac{1}{10}$ normal H_2SO_4 requiring 9.9 cc $\frac{1}{10}$ normal NaOH as this might have been an error in titration. repeated experiment but passed gas into Nesslers Reagent no precipitate showing no ammonia Nesslers is sensitive to 2 cc Nesslers added to 50 cc water contain .05 milligrams Ammonia brown coloration detects free and bound Ammonia

Ni 58.71 Ni SO4 74.68

Exp. #1114

26 Took 10 cc of Ni SO4 Solution from Silver Lake = 1,335 Sp. G. 131 grams Ni per liter = 348 g Ni SO4 or 627 g Ni SO4 7H2O

Requires 7.3

10 cc Na2SO4 Sol + 7 cc NaOH then added Ammonia 10 cc at a time found that it required more ammonia this way than if it is added all at once

Ammonia required = 50 cc of 28% or 93 cc of 15%.

If the ammonia is added first then the NaOH it also requires more ammonia

27 100 cc of Silver Lake Ni SO4 added 70 cc NaOH 20% stirred well then added 930 cc of 15% Ammonia Solution [illegible] slow, precipitation commences within 10 minutes.

28 Rough Experiment

Made some Nickel Carbonate [illegible] with Na2CO3 washing free of Sulphates (BaCl2 test) then filtering off and getting partly dry by standing over night.

Divided this into two parts (approximately) each part dissolved in 200 cc of 28% Ammonia

28 Evaporated in Air in Sluice Room in an evaporating dish, crust formed which when broken looks like large fish scales.

29 Second half of # 28. Evaporated same conditions but in a beaker, less ammonia evaporated a few flat crystals on top.

Amblygonite

1st Hydrofluoric + Sulfuric Method

Big $BaSO_4$ Precip.

Total = 29.5462
Dish = 29.3750
.1712 = 2.26 4

Impossible to wash out the $BaSO_4$ Precip. The greater the $BaSO_4$ precip the greater loss in Li

139122
13.8430
.0692 K_2SO_4
.4487
4844
5536
2768
2768
.031650 04

33.1 % K

29.5030
29.3750
.1280 Na_2SO_4
.3237
8960
3840
2560
3840
04.143360

4.14 % Na

2nd Smith.

29.6320
29.3750
.2570 Li_2SO_4 = 6.984% Li_2O

3rd Acid

Residues from Mr. Edison

37 & #38 1 gram treated by the J. L. Smith process as on page 355 Standard Method of Analysis by Scott. brought down to the Lithium K & Na Chlorides all else precipitated out Mr. Edison tested in the Spectroscope and found just a mere trace of Lithium, said I need not carry them further.

32 Total 29.3746
29.3095
.0651 Li_2SO_4 x .27176

= 1.769% Li_2O

33 Total 57.4260
57.3590
.0670 Li_2SO_4 x .27176

= 1.82% Li_2O

33

Correction by Mr. Edison says he used 10g Lim + 10g Ore so $\frac{10.0000}{20.3648}$ Wt of his residue = 45.9% is

ore x 1.82 Li_2O = 0.835% Li_2O

32

$\frac{10.0000}{20.5285}$ = 48.7% is ore

so 48.7 x 1.769% Li_2O = 0.858% Li_2O

44 TAE

57.4745
57.3590
.1155 Li_2SO_4 x .27176 = 3.138 Li_2O

corrected = 2.98 Li_2O

38.5140
38.4770
.0370 K + Na Sulphates = 1.47% Na_2O

Dissolved in H_2O Filtered on account of insol Residue

38.4830 .1155
38.4770 50
.0060 .1105 x .27176 = 2.98

All corrected for quantity of material used =
Total Wt Residue 21.6065

$\frac{10.0000}{21.6065}$ 46.2% is one

46.2 X 2.98 = 1.37% Li_2O

45 TAE

Total 57.4500
57.3595
.0905 Li_2SO_4 x .27176

= 2.459% Li_2O

Total Weight of Residue 22.127

$\frac{10}{22.127}$ = 45.2% is one

45.2 X 2.45 = 1.1% Li_2O

57.4468
57.3581
.0887 X .27176 = 2.41

47 T.A.E.

57.5162
57.3595
.1567 Li_2SO_4 x.27176 = 4.25% Li_2O

Total Weight of Residue 20,215

$\frac{10.00}{20,215}$ = 49.4% is Ore = 2.09% Li_2O

57.4870
57.3585
.1285 = 3.49

Amblygonite Sulfate Residue Recd Feb. 8/19.
from TAE Total Wt. = 1.7120

S,

Total 7.1080
7.0660
.0420 K_2PtCl_6 = 0.813 % K_2O

Total = 38.7057
38.4753
.2304 x .27176 = 6.26 Li_2O

Total = 38.5400
38.4753
.0647 x .4364 = 2.83 % Na_2O

Test on 4 Oils

Sp. G.

1 Regular Battery Oil	Sp G	@ 20°C	0.9260
2 Straw Oil Regular	" "	" "	0.8725
3 New Straw	" "	" "	0.8715
4 Texas Oil	" "	" "	0.8820
5 Corrus Oil Texas Co	" "	" "	0.8760

Flash Point

1	338°F.
2	300°F
3	300°F
4	364°F
5	345°F.

Viscosity (Reilly)

1	@ 70°F	9 min 36 sec
2	" " "	1 " 11 "
3	" " "	1 " 12 "
4	" " "	3 " 12 "
5	" " "	2 " 6 "

Solidification Test

1	41°F. Sticky	30°F Stiff	22°F Frozen
2	24 " "	18 " "	16 " "
3	22 " "	20 " "	18 " "
4	32 " "	28 " "	22 " "
5	22 " "	18 " "	14 " "

cc	1	2	3	4	5
0	584	538	548	638	604
2	604	546	558	668	614
5	610	560	563	670	618
10	620	580	578	674	624
20	642	600	596	684	642
30	658	612	606		645
40	672	628	626		654
50	683	640	640		652
60		654	650		674
70		672	670		680
80		680	678		
90					

Saponification Number

1	= 6.0
2	= 0.0
3	= 0.0
4	= 0.0
5	= 2.7

Iodine Number

1	= 13.9
2	= 21.8
3	= 21.7
4	= 15.3
5	= 18.7

Acid Figure

1 = 1.22
2 = 0.05
3 = 0.21
4 = 0.24
5 = 0.09

Chemical Preperations

7-inished

1115 Stannous Oxalate 225 grams Stannous Chloride + 25 cc HCl then dissolved in 1000 cc H_2O precipitated cold by pouring a solution of 2940 cc Oxalic Acid into the tin salt. Oxalic Acid content 126 g. washed by decantation until free of chlorides filtered on a funnel filter using a paper filter with a Buchner filter next to the precipitate, so that no paper fibers get into the product dried room temp.

Washed ordinary water but last two decantations distilled Water

1116 Cobaltous Oxalate 7-inished

281 g Cobalt Sulfate in 1000 cc H_2O precip. with 2940 cc of a sol containing 126 g Oxalic Acid, cold, washed by decantation same as 1115 dried in air

1117 Cupric Oxalate Finished
198 g Cupric Chloride dissolved in HCl
the least amount which it took to dissolve it
(as Cupric Chloride is insoluble in water)
I should judge that the water & HCl equaled
about a 15% Sol of HCl.
precip with 126 g Oxalic Acid (2940 cc of
Oxalic Solution, washed by decantation, same
as 1115 dried in air.

Remarks settles rapidly at first but extremely slow when
solid would out extremely fine precipitate
1118 197 g Manganous Chloride in 1000 H_2O
precip by 126 g Oxalic Acid in 2940 cc Sol
H_2O washed, dried, same as 1115
Manganous Oxalate

1119 Calcium Oxalate Finished
111 g Calcium Chloride in 1000 H_2O
precip by 126 g Oxalic in 2940 H_2O Sol.
washed, dried, same as 1115.

Strontium Oxalate Finished
1120 158 g $SrCl_2$ in 1000 cc H_2O
precip by 126 grams Oxalic in a 2940 cc
water Solution, washed, filtered, dried
same as 1115
Made $SrCl_2$ from $SrCO_3$ + HCl.

~~1116~~ 1121 Zinc Oxalate Finished.
136 g $ZnCl_2$
544 cc of a 25% $ZnCl_2$ Sol of Sp. G. 1.238
precip by 2940 cc of the Oxalic Acid Sol
containing 126 g Oxalic Acid
Washed, filtered, dried, same as 1115

~~1117~~ 1122 Cadmium Oxalate Finished
183 g Cadmium Chloride dissolved in 1000 cc H_2O
precipitated with 2940 cc Oxalic Acid Sol
= 126 g Oxalic Acid
Washed, filtered, dried same as 1115.

1123 Strontium) Calcium, Barium Aluminate
Sol of 95 grams Aluminum in 1000 cc 20% NaOH
1123 Strontium Aluminate made by pouring Strontium Chloride dissolved in 1000 H_2O into 1/3 of the Sodium Aluminate Solution

1124 Calcium Aluminate made by pouring $CaCl_2$ dissolved in 1000 H_2O into 1/3 of the Sodium Aluminate Solution

1125 Barium Aluminate
Made by pouring g $BaCl_2$ dissolved in 1000 H_2O into 1/3 of the Sodium Aluminate Solution.

1126. Magnesium Hydroxide
Made by pouring 260 cc of a 36.5 % Solution of $MgCl_2$ Sp. G. 1,306 into 333 cc of 20 % NaOH diluted with 300 cc Water Washed by decantation.

1127 Zinc Hydroxide
Made by pouring 340 cc of Zinc Chloride Solution Sp. G 1,420 into 333 cc 20 % NaOH diluted with 300 cc H_2O washed by decantation

1128 Calcium Molybdate Inverted
98 grams Ammonium Molybdate dissolved in 1000 H_2O + 100 cc Strong Ammonia precip with 55 grams $CaCl_2$ in 1000 H_2O

1129 Barium Molybdate Finished
98 grams Ammonium Molybdate ~~in~~ 1000 cc
H_2O + 100 cc Strong Ammonia precip with
122 grams Barium-Chloride in 1000 cc Water

1130 Strontium Molybdate Finished
98 grams Ammonium Molybdate dissolved
in 1000 cc Water + 100 cc Strong Ammonia
precip. with 79 grams Stront. Chloride in
1000 cc water made from 73g $SrCO_3$ + HCl.

1131 Barium Tungstate Finished
165 grams Sodium Tungstate dissolved in
1000 cc water, precip. by 122 grams
Barium Chloride in 1000 cc Water

1132 Strontium Tungstate Finished
165 grams Sodium Tungstate dissolved in
1000 cc water precip. with 79 grams
Stront. Chloride in 1000 cc water made
from $SrCO_3$ + HCl.

1133 Magnesium Fluoride
Made by precip Sodium Fluoride with 203g
Magnesium Chloride $MgCl_2 6H_2O$ in 1000 H_2O
Sodium Fluoride 2100 cc of 4% Sol.
made from neutralizing NaOH with HFl.

Remarks Very Gelatinous translucent when wet.

1134 Calcium Fluoride
Made by precipitating Sodium Fluoride with 219 g $CaCl_2$ dissolved in 1000 H_2O $CaCl_2$ made from $CaCO_3$ + H_2O

Repeat
1135 Barium Fluoride
Made by precipitating Na Fluoride 2100 cc 4% Sol. with 244 g $BaCl_2 2H_2O$ in 1000 cc water

1136 Zinc Fluoride
Made by precip Na Fluoride 2100 cc 4% Sol with 136 g $ZnCl_2$ 340 cc of a 40% Sol.

1137 Cerium Oxalate (Cerous)
311 grams Cerium Carbonate dissolved in HCl (quite a residue left) filtered precip with ~~[illegible]~~ Oxalic Acid until no more precip.

1138 Strontium Silicate Made by just neutralizing Water Glass so there would be no free NaOH. Method Lunge Vol III page 967 Technische Untersuchungs Methoden. and precipitating with $SrCl_2$ molecular weight in 1000 H_2O
Water glass diluted 6 to 1

1139 Barium Silicate Made by precipitating Water Glass, prepared as in 1138, with $BaCl_2$ molecular weight in 1000 cc H_2O.
Water glass diluted 6—1

1140 Calcium Silicate Made by precipitating Water Glass, prepared as in 1138, with $CaCl_2$ molecular weight in 1000 cc H_2O
Water glass diluted 6—1

1141 Calcium Ferro Cyanide $Ca_2(FeCy)_6$ made by precipitating $CaCl_2$ in 1000 H_2O molecular weight with 5% Excess of Molecular Weight Potassium Ferrocyanide.

1142 Calcium Arsenate made by precip. 219 g $CaCl_2$ in 1000 H_2O with 124 grams Sodium Arsenate in 1000 H_2O

$$2Na_2HAsO_4 + 3CaCl_2 = Ca_3(AsO_4)_2 + 4NaCl + 2HCl$$

1143 Magnesium Fluoride made by precipitating 116 grams Sodium Fluoride in 1000 cc H_2O with 203 grams $MgCl_2 6H_2O$ in 1000 H_2O done in Rubber jar and rubber stirrer.

1144 Nickel Sulphite made by double decomposition Sodium Sulphite and Nickel Sulphate washing the precipitate by decantation

1145 Aluminum Molybdate dissolving Molybdic Acid in NaOH then diluting filtering and precipitating with Aluminum Nitrate washing by decantation

D. rises up to a horny mass brittle with polished fracture

1146 Magnesium Molybdate dissolving Molybdic Acid in NaOH diluting filtering precipitating with Magnesium Chloride washing by decantation.

1147 Zinc Molybdate dissolving Molybdic Acid in NaOH diluting filtering, precipitating with Zinc Chloride washing by decantation

1148 Sodium Antimonite (no proportions)
Made Sb_2O_3 by pouring Antimony Chloride $SbCl_3$ into boiling water 15 times as much water as $SbCl_3$ solution used. washing until neutral $= Sb_2O_3 + SbOCl$ then added water heated on the water bath added a hot solution of Na_2CO_3 to change the $SbOCl$ into Sb_2O_3 just enough Na_2CO_3 to make the it slightly alkaline heated 15 minutes filtered then washed free of Na_2CO_3 the Sb_2O_3 was then dissolved in NaOH hot diluted and filtered.
Tested by adding a little HCl passing H_2S orange precip of Antimony Sulphide

1149 Sodium Telluriite made by dissolving Tellurium in Aqua Regia poured into water = H_2TeO_3 Tellurous Acid; washed free of chlorine then dissolved immediately in NaOH hot.
the precipitated H_2TeO_3 must not stand long as TeO_2 will crystallize out.
Tested by dissolving in dilute H_2SO_4 adding KI sol. precipitates Tellurium Iodide which redissolves in excess of the reagent. (test for Telluriites)

1150

Analysis of Recovered Ni from Ni used from S. B. Co given to me by Mr. Edison July 18, 19.

No 1 10 cc

Total = 15.0270
Filter + Bottle = 14.9700
Ag Cl .0570 X .25435 = .014497 HCl
say .0145 X 10 X 37.85 = 5.488 HCl per gallon

Total Sulphates 5 cc

Total 10.2405
Crucible 9.1228
1.1177 $BaSO_4$
20
22.3540 $BaSO_4$ X .42020 = 9.4931 g H_2SO_4
per 100 cc X 37.85 = 359.31 grms H_2SO_4 per gallon

Iron & alumina 25 cc

12.5700
12.5610
.0090
.0360 X 37.85 = 1.36 g $Fe_2O_3 + Al_2O_3$
per gallon

No 1

Copper 25 cc

.4 cc x 4 1.6 for 100 x 37.85 = 60.56 cc per gal

.466 grams Cu per gallon

$\frac{4.66 \times 98}{63.57}$ = .7183 H_2SO_4 taken up by the copper to form $CuSO_4$

Nickel

319.91 − .7183 = 319.191 H_2SO_4 to form $NiSO_4$

$\frac{319.91 \times 58.68}{98}$ = 191.12 grams Ni per Gallon

Free Acid

10 $\frac{N}{2}$ H_2SO_4 require 10.1 NaOH 10 cc taken

50 cc $\frac{N}{2}$ NaOH made up to 250 cc took 25 cc

required 1.5 cc H_2SO_4 took 1.5 x 10 = 15 cc

49.5 − 15 = 34.5 x .0245 = .84525 x 10

= 8.4525 H_2SO_4 per 100 cc to make Ni & Cu sulphates. 9.493 total sulphates − 8.452

= 1.041 H_2SO_4 x 37.85 = 39.3 g H_2SO_4 as Na Sulphate

319.91 H_2SO_4 per gallon to form Ni & Cu SO_4.

$\frac{39.3\ H_2SO_4 \times 142}{98}$ = 57.16 Na_2SO_4 per gallon

Free Acid 0.0

Ni from Ni Mud #2

10 cc

Total = 13.3720

Filter & Bottle = 13.3433

.0287 x .25435 = .007299

x 10 = .073 x 37.85 = 2.76 g HCl

Total Sulphates 5 cc

10.0105

9.1975

.8130 $BaSO_4$

20

16.2600 $BaSO_4$ x .42020 = 6.832 grams H_2SO_4

per 100 cc x 37.85 = 258.59 g. H_2SO_4 per gallon

Iron and Alumina 25 cc

12.5785

12.5700

.0085

4

.0340 x 37.85 = 1.286 g Fe_2O_3 + Al_2O_3 per gallon

No 2

Copper 25 cc

25 cc took .5 cc KCn .5 X 4 = 2. X 37.85

75.70 X .0077 = 0.582 g Cu per gallon

$\frac{.582 \times 98}{63.57}$ = .897 H_2SO_4 to form $CuSO_4$

Nickel

255.01 − .897 = 254.113 H_2SO_4 to form $NiSO_4$

$\frac{254.113 \times 58.68}{98}$ = 152.15 Ni per gallon.

By Electrolysis 32.4745 / 32.0780 / .3965 .3965 for 10 cc x 10 = 3.965

x 37.85 = 150.07

Nickel Hydrate Titration

Took 10 cc 50 cc $\frac{n}{2}$ NaOH up to 250 cc back

25 cc required 2.2 cc $\frac{n}{2}$ H_2SO_4

2.2 x 10 = 22 cc for 100 cc solution

Since 10 cc $\frac{n}{2}$ H_2SO_4 requires 10 cc $\frac{n}{2}$ NaOH 50 − .5 = 49.5 cc

10 cc actually used 49.5 − 22.0 = 27.5 cc used in

changing the Ni & Cu to Hydrates 27.5 X .0245

= .67375 x 10 = 6.7375 g H_2SO_4 for 100 cc solution

6.7375 x 37.85 = 255.014 g H_2SO_4 per gallon

258.59 − 255.01 = −3.58 H_2SO_4 as Ni Sulfate!

$\frac{3.58 \times 142}{98}$ = 5.18 grams Na_2SO_4 per gallon

Free Acid

O.O.

July 19.19.

41 lots of Nickel active Material from inside of old tubes put in Hyposulphite of Soda.

Proportion

Same as in big batch of nickel tubes. Page 95

Sol. made in 1914.

Big Batch	On Each Series
4000 C.C. Water	352
1200 grams Hyposulphite Soda	106.
2265 grams (5 lb) Material	200

Sp. G = 1138 at 13° C

On July 19, 1919 at 11.30 A.M.

Temp. of solution 26° C

Rise " " " to 40° C

Did not disintegrate more than 80%

Copper Wash Water from 5.13.60
Copper by the Parkes Cyanide Method.

100 cc require 6.1 cc
1 cc of Cyanide = .0077 Cu
6.1 × .0077 = .04697 g Cu in 100 cc × 37.85
= 1.777 grams Cu + Ni per gallon

Copper precip. by H_2S when electrolyzed
32.0950
32.0790
.0160 Cu in 100 cc × 37.85 = 0.605 g Cu per gallon.

Cyanide Method 2.6 × .0077 × 37.85 = .75 Cu
43.7585
43.6440
Dimethylglyoxime Ni .1145 × .2032 = .0233 Ni in 100 cc × 37.85 = .8819 g Ni in a gallon

Ni Wash Water

Cu by Parkes Method 100 cc passed H_2S
washed dissolved HNO_3 + NH_4OH required

11.7
11.0
.7 cc KCN 1 cc KCN = .0077 Cu

X .7 = .00539 Cu X 37.85 = 0.204 Cu

per gallon

Total	43.6915	
Glass + Filter	= 43.5036	
Ni Dimethyl	.1879	X .2032

X 37.85 = 1.445 grams Ni per gallon

3

Copper

25cc required 27.2 − 7.0 = 20.2cc KCN × 4 = 80.8cc
× 37.85 = 30582.8 × .0077 = grams Cu per gallon
= 23.548 grams Cu per gallon
23.548 × 98 / 63.57 = 36.3 grams H_2SO_4 per gallon to form $CuSO_4$
Total Sulphates

Total = 44.9430
44.5710
.3720 for 5cc
20
7.4400 $BaSO_4$ per 100cc

7.44 × 37.85 = 281.6 $BaSO_4$ × .42 = 118.272 H_2SO_4 per gallon.

Iron & Alumina as Fe_2O_3 Al_2O_3 25cc.

Total 44.5870
44.5710
.0160 × 4 = .0640 × 37.85 = 2.42 grams per gallon.

H.Cl good trace

Titration.

10cc to 25.0 leaves 25cc required 24.9 − 21 = 3.9cc
back 3.9 × 10 = 39cc Total 50.5 = 50cc
50 − 39 = 11cc for 10cc solution used to form
Ni Cu Hydrates. = 11 × .0245 = 0.2695
× 10 = 2.695 H_2SO_4 for 100cc × 37.85 = 101.9 grams
H_2SO_4 used up in forming Ni & Cu Sulphates.
118.272 − 101.9 = 16.37 grams H_2SO_4 left to
form Na_2SO_4 = 16.37 × 142 / 98 = 23.73 grams
Na_2SO_4 per gallon.

101.9 − 36.30 = 65.6 = grams H_2SO_4
for the Ni = 65.6 × 58.68 / 98 = 39.2 grams Ni per
per gallon

Free Acid None

Nickel by electrolysis.

32.1465
32.0790
.0675 for 75cc .0675 / 75 = .0009 for 1cc
× 250 = .225 for 25cc × 4 = .9 grams per 100
× 37.85 = 34.065 Ni per gallon

1148

Treatment of Ni from Old Tubes

225 grams Old Tubes
352 cc Water
120 grams C.P. Sodium Hyposulphite.
On 96 hours.
then screened thru 10 mesh screen
43 grams or 19.1% remained on screen.

200 grams Old Tubes
352 cc. Water
106 grams Hyposulphite Soda from C. Cooper.
on 96 hrs.
Screened thru 10 mesh
61 grams or 30.5% remains on screen.

200 grams Old Tubes
352 cc Water
132.5 grams Sodium Hyposulphite (Cooper)
On 96 hours
Screened thru 10 mesh
66 grams or 33.0% remained on screen

200 grams Old Tubes
352 cc Water
159 grams Sodium Hyposulphite (Cooper)
on 96 hrs.
Screened thru 10 mesh
58 grams or 29.0% Remained on Screen

200 grams Old Tubes
352 cc Water
212 grams Hypo sulphite Soda (Cooper)
on 96 hrs
Screened thru 10 mesh
58 grams or 29%. remained on Screen.

Old Ni Pocket Material washed free NaOH
dried 4" above hot plate.
200 grams this material
500 cc 15.2% Sodium Sulphite Sp. G. 1.153 @ 60°F
on 4 hrs
Screened thru 10 mesh.
64 grams or 32% remained

Ni from old pockets washed free NaOH
dried 4" above hot plate
took 201 grams this material
500 cc 32.88 % Sodium Hyposulphite ($Na_2S_2O_3$)
Sp. G. @ 60°F. 1306
on 48 hrs
Screened thru 10 mesh
62 grams or 31 % remained on screen.

Selenium Analysis Aug. 12/19.

15.4369
14.9559
.4810

1st Sol'n made for this Solution to use in Vat Wash
10 cc Taken
Precip Se with SO_2
found .481 grams Se
per Liter

Big Batch. Aug. 12, 19

19.8750
14.9582
4.9168

1147.5 grams Se dissolved up
then made up to 2000 cc
10 cc Taken precip Se with
SO_2 = 491.68 grams per Liter
= .49168 grams per cc made up to 20 Liters

2 liters add 18 liters
1 " " 9 "
100 cc " 900 cc

{ 200 cc Sol + 1800 cc Water.

Aug 13/19.

Time Test on Settling of Electrolytic NaOH.

Rolled Drum to get the NaOH dirty they drawn out into a 1000 cc graduate on 11.00 AM Aug 13, 19.

Aug 14	Electrolyzed	50 cc	Quite dark deposit
Aug 16	"	50 cc	Slight Dark Deposit
Aug 18	5 days "	"	Trace -

Method for Checking
Copper, Nickel, and Selenium Bath Solutions

Method for Selenium Solutions

For solutions containing 50 or more grams per Liter take 10 cubic centimeters

Exp. on Old Ni Pockets

50 grams Ni from Old Pockets
200 cc Water
12.5 grams Anhydrous Glucose
10 cc 20% NaOH
Put on butter dish on hot plate Aug. 15, 10:45 AM

50 grams Ni from Old Pockets
200 cc Water
25 grams Anhydrous Glucose
10 cc 20% NaOH
Put on butter dish on hot plate Aug. 15 10:45 A.M.

50 grams Ni. from Old Pockets
200 cc Water
50 grams Anhydrous Glucose
10 cc 20% NaOH
Put on butter dish on hot plate Aug 15, 10:45 AM

Aug 19/19

Nickel Chloride Solution

Nickel Hydrate dissolved in H.Cl (commercial)
Sp. G. of Sol. 1,301 @ 23° C.
Took 5cc diluted to 100cc and took 5cc of this.

15.4217 Total
15.1902 Glass
.2315 Precip. X .2032 = .04704080
X 20 = .94081600 X 20 = 18.816 g Ni per 100 cc

contained copper which precip with the Dimethylglyoxime

Nickel Chloride Solution
Sp. G. @ 23° C 1310

Wt = 60.0464 − 59.9098 = .1366 Ni per 1 cc = 13.66 Ni in 100

G Electrolysis

G Cass = 15.8779 − 15.2045 = .6734 X .2032 = .1367 Ni in 1 cc

= 13.67 Ni in 100 cc Solution G Dimethylglyoxime Method

Nickel Silicofluoride Solution

708 grams Nickel Carbonate (Cooper) dissolved in 2880 cubic centimeters of 1275 Sp. G Hydrofluosilicic Acid = 30%
kept cool by running water max temp. = 35° C
filtered, this solution left a lot of silica back on the filter.

Total volume = cc

$NiCO_3$ = 118.

Experiment to determine Plating Value and Acidity curves of Sat. Sol $NiSO_4$ + 6 g Magnesium Acetate per 250 cc

Exp 1 Copy Mr. Edisons Note

Make up enough solution and some to spare to fill 6 tumblers with 250 cc of $NiSO_4$ Conc and containing 6 grams each cell of Acetate of Magnesia

No each cell 1A 2A 3A 4A 5A 6A

Use single strip nickel anode & neg felted varnished Cathode same as we have been using

Use 400 Milliamperes C.D. instead of 200 as we have been using

Take out of circuit 1A after it has run 2 hours 2A 3 hours etc

I expect the longer the cell runs the more acid it will get. I want you to take out a little and determine the acidity rather accurately of each seperate cell this will show a curve of increase acidity as plating is prolonged and is due to small surface of anode

Exp. 2

Now take the solutions of Exp 1 and to each seperately add freshly precipitated Ni hydroxide wash almost free of NaOH & Sulphates and neutralize each cell and filter.

I will have a compound anode made of 6 strips so as to have 6 times the surface of anode as in Exp. 1

These 6 cells are to be run with 400 m.a. and marked 1B 2B etc — 1B comes off in 2 hours. 2B in 3 hrs etc.

You are then to determine the acidity of each cell seperately if it shows very much less acidity it will prove our single anode is entirely too small

Exp. 3 Take the 6 cells from exp. 2 after having determined acidity & neutralize each seperately with $Ni(OH)_2$ and run them exactly the same as in exp. #2 using the compound anode of 6 plates you should after all are removed determine acidity again neutralize continue this until I tell you to stop which I can tell by

the quality of the plating. There many runs will show if the acidity is changed.

Exp. 4 Make up 6 fresh cells as in Exp. 1. Run them just like # 2 with multiplate anodes but instead of 400 milliamps use 800 milliamps & determine acidity

You will be able with these various acidity determinations to plot them on a curve sheet after above exp. I will tell you what to do with them.

Exp. # 1

1A off in 1 hour
2A " " 2 hours
3A " " 3 hours
4A " " 4 "
5A " " 5 "
6A " " 6 "

} C.D. 400 milliamps.

Acidity Tests.

Value of $\frac{N}{2}$ NaOH 1^{cc} = .02519 grams H_2SO_4

used 5^{cc} for test

No	cc of $\frac{N}{2}$ NaOH	GRAMS H_2SO_4 per 100^{cc} sol.	GRAMS ACETIC ACID PER 100^{cc} SOL
Original Solution	0.2	0.10076	0.120
1 A	0.6	0.30228	0.360
2 A	1.0	0.50380	0.60
3 A	1.4	0.69532	0.84
4 A	2.0	1.00760	1.20
5 A	2.3	1.15874	1.38
6 A	2.8	1.41064	1.68

Test of $NiSO_4$ Sol-C...

$NiSO_4$ Sol from Tudell contains more trace of copper. and .0324 grams Fe_2O_3 per 100 cc

The Solution from 22 Building (Special Bath run for Mr Edison) contains.

Experiment #2

Continuation of Exp. 1 Page 117

After Neutralizing with $Ni(OH)_2$ 5 cc Sample

1 B	0.1 cc	= 0.0503	grams	H_2SO_4	per 100 cc	
2 B	0.1 cc	= 0.0503	"	"	"	
3 B	0.1 cc	= 0.0503	"	"	"	
4 B	0.1 cc	= 0.0503	"	"	"	
5 B	0.1 cc	= 0.0503	"	"	"	
6 B	0.1 cc	= 0.0503	"	"	"	

After Running at 400 M.a. with Anodes of 6 lines the Surface

5 cc Sample	cc $\frac{N}{2}$ NaOH	Grams free H_2SO_4 per 100 cc
1 B	0.5	0.2519
2 B	0.9	0.4534
3 B	1.8	0.9068
4 B	2.0	1.0076
5 B	1.9	0.9572
6 B	2.2	1.1083

Experiment Making Alkaline Nickel & Copper Solutions.

10 cc Sat. Sol Copper Sulphate + some Sodium Citrate then added 20% Na OH Precipitate the Cu when a large excess of Na OH is added

10 cc Sat. Sol Copper Sulphate + some Sodium Citrate + excess of Na OH remains clear.

10 cc Sat. Sol $NiSO_4$ 1.300 Sp. G. + 1/2 gram Na Citrate 2 cc Na OH 20% to neutralize then added 2 cc excess did not dissolve the free $Ni(OH)_2$

10 cc $NiSO_4$ + 1.5 g Na Citrate 2 cc Na OH 20% to neutralize + 2 cc extra did not dissolve the free $Ni(OH)_2$

10 cc $NiSO_4$ + 2.5 g. Na Citrate 2 cc Na OH to neutralize + 2 cc extra formed jelly insol in excess Na OH

10 cc $NiSO_4$ + 3 g Na Citrate + 2 cc Na OH + 2 cc extra No $Ni(OH)_2$ precip. occurs. clear on standing jelly formed in about 1 hour clear jelly.

5 10 cc $NiSO_4$ + 5. grams Na Citrate + Solid of 4 cc Na OH No $Ni(OH)_2$ precip forms did not jelly in 3 hours but if a little More Na OH is added goes solid.

6 10 cc $NiSO_4$ + 10 g Na Citrate took 2 cc to neutralize ? (as the reaction apparently takes a long time to take place) go solid). added immediately 5, 6, 7, 8, 9, 10, 11, 12, 13, 14 to 20 cc 20% Na OH when it went solid as a clear jelly in a few seconds. then adding about 20 cc More Na OH sol. it goes to the usual colored $Ni(OH)_2$ precipitate.

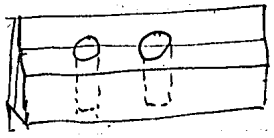
10 cc Sat. sol $NiSO_4$ + 1/2 gram Sodium Tartrate + 16 cc 20% Na OH Precipitated $Ni(OH)_2$ does not dissolve.

10 cc Sat. Sol $NiSO_4$ + 1 gram Sodium Tartrate + 16 cc 20% Na OH just a little precip which clears up in about 2 hours.

10 cc Sat. Sol. $NiSO_4$ + 1 1/2 grams Sodium Tartrate 6 cc Na OH to Neutralize + 6 cc extra = 12 cc 20% Na OH in all sol remains clear. 20% Na OH can be added ad libitum without giving a precipitate (can not plate from it)

1150

Experiment on Making Cl free KOH

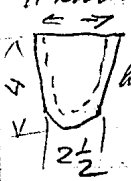


Hole 1" X 4 3/4
Block 4" X 10 1/2
X 4 3/4 6 Holes
covered over to keep out CO_2

200 grams KOH from Silver Lake marked (Sample of Solid KOH Niagara Alkali Co 2.19% KCl used for making electrolyte.)
+ 10 grams KCl melted in an iron retort and poured into the mould, mould so hot nearly all boiled over, waited until cooler then filled up last about 1/2" which boiled over time 10.30 at 11.00 just solid on top do not think it was solid all the way through., let it cool naturally by taking off the fire,

Heated 2.93 grams KOH + 8.2 grms HCl as the KOH contained 6.4g HCl the made 14.6 or 5% KCl added.

Heated to boiling in an iron retort then banked the fire filling hot ashes all round put on lid tested from time to time took from 11.30 AM to 3 H 5 P.M. to form a crust on top. let stand till morning then tapped pot with hammer would not break loose, heated pot as much as I dared without melting KOH and turned over and tapped did not come out so I made a borer of pipe to drill it (core drill) bored in three samples Top, Middle, Bottom.



Top. KCl	4.93	Green
Middle	5.312	Light Yellow
Bottom	3.95	Black

Bottom has a lot of precipitated Iron.

1151

Exp. Taking Manganese out of Iron by Electrolysis.

#1 25 cc of a saturated solution of Manganous Sulphate + 75 cc Water C.D. 100 M.a. 3 min Dark Chestnut deposit on anode.

Sol #1 + 1 cc Ferrous Sulphate Sat Sol. C.D. 100 M.a. 3 min Dark Chestnut deposit on anode

Kept adding Ferrous Sulphate up to 50 cc when there was no deposit on anode except a dark line at the point of contact with the electrolyte

Tried the same with 10 cc Ferrous Sulphate using a porous cup to seperate Anode no improvement

Also tried Sat. Sol Ferrous Sulphate + 10 cc Sat Sol Manganous Sulphate 50 – 70°C C.D. 100 M.a. and C.D. 600 M.a. Just a line of deposit on anode at the point of contact with the electrolyte. Added 25 cc 60% Acetic Acid according to Smith No change.

Ferric Solutions

The Solution used in the following experiments was Sat Sol of Ferrous Sulphate changed to Ferric Sulphate by adding just enough H_2O_2 Tested with Ferricyanide to see that all was converted into Ferric

Solution of Mangani Sulphate saturated.

1 25 cc Manganic Sulphate + 75 cc Water C.D. 100 M.a. Time 3 min Dark Brown Deposit nearly Black all over Anode.

2 Same sol as #1 except added 1/2 cc of Sat Sol Ferric Sulphate C.D. 100 M.a. Time 3 min. Dark brown nearly black deposit on anode even all over.

3. Same sol as #1 except 1 cc Sat sol Ferric Sulphate. C.D. 100 M.a. Time 3 min Dark brown nearly black deposit on anode even all over

4 Same sol as #1 except 2 cc Sat. sol Ferric Sulphate C.D. 100 M.a. Time 3 min Dark brown nearly black deposit on anode even all over.

5 Same sol. as #1 except 5 cc Ferric Sulfate C.D. 100 M.a. Time 3 min Dark brown nearly black deposit, even all over anode.

6 Same sol as #1 except 10 cc Ferric Sulfate. C.D. 100 M.a. Time 3 min. Deposit all over anode but of a medium brown.

7 Same Sol as #1 except 15 cc Ferric Sulfate C.D. 100 M.a. Time 3 min Deposit on anode in spots of a light brown.

8 25 cc Manganic Sulfate + 75 cc Water + 20 cc Ferric Sulfate Anode put in porous cup. C.D. 100 M.a. Time 3 min. Medium brown deposit all over anode

9 Same Sol as #8 except 25 cc Ferric Sulfate C.D. 100 M.a. Time 3 min Anode in porous cup. Just a light straw deposit let it go for 10 min deposit very little darker.

10 100 cc Ferric Sulfate + 10 cc Manganic Sulfate. Porous cup around Anode C.D. 100 M.a. Time
3 min trace of light deposit straw color
17 " Just a little darker
30 " No additional Deposit.

11 Same as #10 except temp. raised to 50° C
Time 3 min. Light Brown Deposit
" 15 min No darker than at 3 min.

12 Same as #10 except temp raised to 70° C no difference from #11

13 added to #10 after cooling down to room temp. 20 cc H_2O_2
C.D. 100 M.a.
Time 3 min Slight deposit on anode of a light straw color added 5 cc More H_2O_2 in the Anode compartment
C.D. 100 M.a Time 3 min not a trace of deposit
C.D. " " " " 15 " " " " " "

1152

Seperating Mn from Fe by crystillization

Original Solution weighed on rough scale

450 g $FeSO_4$ + 50 g $MnSO_4$ to 1000 cc

10 cc dil to 200 cc took 20 cc for analysis

Mn in original sol. 12.5660

12.5436

Mn_3O_4 = .0224 g × 0.720 = .01612

g Mn in 20 cc or in 1 cc original sol 100 = 1.612 g Mn in 100 cc sol

× $\frac{1.612 \times 223}{5493}$ = 6.50 g $MnSO_4 4H_2O$ in 100 cc

= 65 g $MnSO_4 4H_2O$

Manganese in Mother Liquor

10 cc dil to 200 cc took 20 cc for analysis

12.5566

12.5436

Mn_3O_4 .0130 g × 0.720

= .00936 g Mn in 1 cc

1 g to 200 cc took 20 cc for analysis

Mn in 1st cryst. 12.5450

12.5436

Mn_3O_4 in 1 g .0014 × 0.720 = .001 g

Mn × 10 = .01 g Mn in 1 gram $FeSO_4 7H_2O$

10 cc to 200 cc took 20 cc for analysis

Fe in original sol. 12.6840

12.5436

Fe_2O_3 .1404 × .7 = .09828

.09828 × 100 = 9.828 g Fe in 100 Sol.

9.828 × 4.979 Fe to = 48.93 g $FeSO_4 7H_2O$

= 489.3 g $FeSO_4 7H_2O$ in the total 1000 cc Sol.

$\frac{1.000}{.09828}$ = 10.17 × .0161 = Mn in each 20 cc

= .164 g Mn per 1 g Fe = 16.4 % Manganese

10 cc to 200 took 20 cc for analysis

Fe in mother liquor 44.6626
44.5720
Fe_2O_3 .0906 x .7 = .06342 g.

Fe in 1 cc mother liquor = 6.342 g Fe in 100 cc

$\frac{1.000}{.06342}$ = 15.7 x .00936 = .1469 Mn

per 1 g Fe = 14.69 % g difference

11.78 Mn.

1 g Crystals to 200 cc took 20 cc for analysis

Fe in 1st crystals. 12.5745
12.5436
Fe_2O_3 .0309 = .02163 Fe

in 1 g Crystals or 20 cc Solution:

$\frac{1.000}{.02163}$ g Fe = 46.2 x .001 g Mn in 20 cc

= .0462 g Mn in 1 g Fe or 4.62 % Mn contained in each gram of Iron (Metallic) in the Iron Sulphate crystallized out.

1152

took 100 cc Original Solution of 1000 cc which contained 489.3 g $FeSO_4 7H_2O$ + 65 grams $MnSO_4 4H_2O$

Put into a beaker placed in salt and ice under constant stirring filtered rapidly when 3 ° below 0 °C. washed crystals on filter with about 200 cc Sat. Sol Ferrous Sulphate (no Mn) dried in air. Vol of Mother Liquor 110 cc

Exp 1152

Stock Solutions

Ferrous Sulfate (Sat.) + Manganous Sulfate

Total = 44.7020
Crucible = 44.5720
.1300

Fe_2O_3 = .4526 $FeSO_4 7H_2O$ or .091 g Fe
per 100 cc = 45.26 g $FeSO_4 7H_2O$ per 100 cc Sol.

Total = 12.5657
Crucible = 12.5436
.0221

Mn_3O_4 = 0.0159 g Mn
per 1 cc = .064 g $MnSO_4 4H_2O$ or 6.4 g $MnSO_4 4H_2O$ per 100 cc
For analysis 10 cc Stock Solution to 200 took 20 cc of this. = 19.08 g Mn per 1.0 g Fe

Ferrous Sulfate No Manganese (Sat. Sol)

Total = 12.6910
Crucible = 12.5436
.1474

Fe_2O_3 = $FeSO_4 7H_2O$ or .1031 g Fe
per cc.

1 g Fe = .2032 Gravimetric.
for every 100 g Fe 20.32 g Mn or 16.8% Mn
Figuring as Metal = 83.2 Fe
16.8 Mn

Exp 1152

#1 200 cc of Stock Solution evaporated in porcelain dish 24 hours under agitation from time to time Room Temperature
Filtered. 35.8 grams crystals.
Ferrous Sulfate with 120 cc Mother Liquor crystals Not washed. Big Crystals.

	Mn	Fe	Mn per g Fe
Mother Liquor			
1st crystals (per 10 grm) →	.0029	.02457	0.120 = 10.7%
Mother Liquor from Recryst 1st Cryst.			
Recryst. of 1st cryst. 1/10 g bottle	.0015	.0223	.067 6.2%

For analysis 1 g Crystal to 200 took 20 cc
Dissolved 1st crystals 34.8 grams in 70 cc Water = 85 cc. Sol. Let Stand 24 hours
got Large Crystals 16 grams which I ground and mixed for an average for analysis
40 cc of Mother Liquor
Losses 1 g lost by running on dish which could not be scraped off 1st cryst
Original Figures 1st cryst. .0351 Fe_2O_3 per 10 g cryst.
" " .0041 Mn_3O_4 " " "

#3. 500 cc Stock Solution brought down to −3° C under constant stirring (salt and ice) then rapidly filtered on vacuum filter crystals not washed 108.6 g Crystals 430 cc Mother Liquor. 1 g Cryst. for Analysis

Dissolved 1st crystals in 170 cc H_2O total volume 231 cc Sp. G. 1265. Room Temp. Recryst. at −3° C
g. Crystals 206 cc Mother Liquor.

	Mn	Fe	Mn per 1 g Fe
1st Crystallization per 10 g cryst.	.00612	.03227	~~.1896~~
Mother Liquor 1st Cryst.			
Recryst. of 1st Crystals	.0014	.0214	.064
Mother Liquor from Recrystl 1st cryst.			
1st cryst. Fe_2O_3 per 1/10 g Crystals	.0461		
" " Mn_3O_4	.0085		

#2 500 cc Stock Solution put into a 600 cc Beaker placed in ice box Nov. 10/19 at 11.20 A.M. temp. 10° C. taken out Nov 12 11.20 AM Temp. 4° C Time 48 hours. Drained off Mother Liquor washed crystals in beaker with 25 cc Sat Sol Ferrous Sulphate no manganese drained dried in air.
= 75.5 grams crystals
Mother Liquor 470 cc − 25 cc = 445 cc

For 1/10 g Salt. .0012848 Mn
" " " .020128 Fe
For 100 g. Fe there are 6.38 g Mn.

#4 500 cc Stock Sol brought down to −3 °C with Ice and Salt under constant stirring kept. 10 min at −3 then filtered rapidly vacuum filter took of 1 gram crystals for analysis. then washed the rest while still on the filter with 100 cc Sat. Sol Ferrous Sulfate No Manganese sucked dry dried a little in air and weighed

Crystals 96 grams. Crystals extremely fine

Mother Liquor 545 cc − 100 = 445 cc

	Mn	Fe	Mn per 1 g Fe
Crystals unwashed $\frac{1}{10}$ g	.0016	.0198	.083
" washed	.0016	.0198	.082

	Mn Volumetric	Gravimetric
Crystals unwash $\frac{1}{10}$ g	.0015257	.001656
"	Fe "	Fe "
" " $\frac{1}{10}$ g	.019584	.01988

Dissolved 9.6 g crystals in 200 cc Water recryst. under same conditions as 1st time except did not wash crystals Crystals 24.7 g Mother Liquor 216 cc.

Fe for $\frac{1}{10}$ g crystals .02074

Mn " " " .001004

7 gr every 100 grams Iron in the crystals

4.84 grams Manganese

For 3rd cryst. see page 141

#5 500 cc Stock Sol brought down to −3 °C with ice and salt without stirring remained in ice and salt 2 hours. then filtered on vacuum filter. took off 1 gram for analysis washed rest with 100 cc Sat Sol Ferrous Sulfate (no manganese) while on the filter then sucked dry, dried in air weighed

Crystals 1/8 to 1/4" size 64 grams

Mother Liquor 560 cc − 100 = 460 cc

	Mn	Fe	Mn per 1 g Fe
Crystals unwashed $\frac{1}{10}$ g	.0016	.0204	.0813
" washed	.0015	.0195	.0774

	Mn Gravimetric	Mn Volumetric
Crystals unwashed $\frac{1}{10}$ g	.001656	.001606
Difference	.00005	

between Gravimetric & Volumetric

#4 Took the crystals from recryst. of the 1st crystals and dissolved in least quantity water recryst. same as 1st cryst.

For each 10 gram crystals Fe in the crystals there are .0003212 Mn.

Fe for 10 g crystals .023936

For each 100 g Fe in the crystals = 1.34 g Mn.

	mn.	% drop
Original for every 100 g Fe in Crystals.	20.32	59.2
1st cryst	8.3	41.7
Recryst 1st crystals	4.84	72.4
2nd recrystallization	1.34	

Figuring the average drop in Manganese per crystallization to be 50%
it would take 7 cryst. to bring it down to .04 g Mn per 100 g Fe in the crystals

#6 Glauber salt solution cooled down to 0°C filtered rapidly giving a mother liquor saturated at 0°C

Cooled 200 cc of this mother liquor + 5 cc H_2O added down to 0°C and poured in under constant stirring 100 cc Stock Solution of Fe + Mn.
No crystallization took place but after 10 minutes a large quantity of ice was formed temp going down to −4°C as this did not happen when the Stock solution was used straight, it shows that either the double salt is more soluble or that the dilution was too great.

#7 50 cc of Glauber Salt Mother Liquor sat at 0°C cooled down to 0°C poured 150 cc of Stock Fe Mn Sol into this under constant stirring cooled down to −4°C
Salt. 10.5 grams
Mother Liquor 194 − 50 = 144.
For 1/10 gram Salt. Fe .019856
" " " " Mn .001525
= For every 100 grams Fe in the salt there are 7.68 grams Mn.

135 [illegible] 105
$ZnSO_4$

8 100 cc Stock Iron & Mn Solution poured into 50 cc of a Solution of Zinc Sulphate which was saturated at 0°C poured in while Sol was at 0°C constant stirring obtained 16 g of crystals (Filter Vacuum Filter).
Tested for Zinc & precip Fe with NH_4OH having an excess NH_4Cl in the Fe Solution then zinc in acetic Acid Sol. with H_2S. Much zinc in the crystals.
100 H_2O dissolves 43 g $ZnSO_4$ @ 0°C

9 100 cc Stock Iron & Mn. Solution poured under constant Stirring into 30 cc of Zinc Chloride Sol Sol. at 0°C. Temp 0°C filtered vacuum filter
34 g Crystals. 105 − 30 = 75 cc Mother Liquor

Tested crystals as in # 8 for Zinc much zinc in the crystals
100 H_2O dissolves 209 g $ZnCl_2$ @ 0°C

10 100 cc Stock Iron & Mn Solution poured into 50 cc of Na NO_3 Solution Saturated at 0°C constant Stirring Temp 0°C when poured in 13 gram Crystals contain much Manganese (Qualitatively).

8 Recrystallized 1st crystals
10 g Crystals Fe = .01635
" " Mn = .0009636
For 100 g Fe in crystals 5.90 grams Mn.
Qualitatively it shows too much Zinc remaining in the Crystals.

9 Recryst. 1st crystals
10 g Crystals Fe = .017408
" " Mn = .0009636
For 100 g Fe in crystals = 5.53 grams Mn
Qualitatively too much zinc remaining in crystals.

10 grams Sample of Crystal. Stock Solution contains for 100 grams Fe in Crystals 9.45 Mn

1 cc of 1 to 9 Nitric Acid added roasted temp. (not known) leached with Hot water free of Sulphate — insol 1.3955 for Every 100 grams Iron .41 g Mn.

Melting Point on Samples of Roffers Resin (Cumarone)

Four Pasteboard Boxes in Training School Marked 1, 2, 3, 4, Two boxes in Lab Marked 5, 6. and One Barrel.

Sample	°C	°F
Sample from Big Barrel.	75-76	167-169
" # 1	95-98	203 - 208
" # 2	30-42	86 — 108
" # 3	96-98	205 - 208
" # 4	96-98	205 - 208
" # 5	96-98	205 - 208
" # 6	100-101	212-214

6 is the one used first.

Ferrous Sulphate & Manganese Sulphate (Stock Solution)
added H_2SO_4 then oxidized with HNO_3
added Na_2CO_3 until just about neutral then about
2g Na_2SO_4 for every gram iron, boiled,
precipitate of Fe(OH)$_3$ as Basic Ferric Sulphate

very hard to filter comes down somewhat like a
jelly, boiled for 1 hours, practically no
change in its filtering quality.
solution acid.

Analysis Trace of Manganese in the Iron
Precipitate
The liquid above the precipitate is quite
white when thrown on the filter it is so
finely divided and colloidal like that it
will run thru the filter this I found
to be dissolved up to a clear liquid in
the morning boiled a little first
part of the iron precipitated, I tried another
portion with H_2O_2 boiled No precip.
still another portion neutralized with
Na_2CO_3 boiled all came down supernatant
liquid clear white, but turned yellow
in one hour.

Ferrous & Manganous Sulphate (Stock Solution)
added H_2SO_4 then oxidized with HNO_3 made
nearly neutral with Na_2CO_3 then added
boiled, precip. [illegible] Na_2CO_3
little at a time until there was only a little left
left in solution filtered.

drainings of the Iron Carbonate precipitate
contains not even a trace of Manganese.

Ferrous & Manganese Sulphate crystals from Steels Solution were put into a sealing jar [illegible] Bailey bar for a Muffle and heated as follows passing in a current of air.

[illegible]	[illegible]	Sample
12.00	105	
1.30	120	I
2.30	148	
3.50	150	II
4.50	195	III
Next day 8.50	Room	
[illegible]	250	
10.10	250	IV

Adding water to
#1 does not heat up does not calce up sol clear all dissolves up [illegible] if filtering is [illegible] [illegible] all of [illegible]
#2 same as #1
#3 heats up slightly calces up
#4 heats up quite a little much fines
Some Ferric.
All Four dissolve.

Heated another bottle for 7 hours at 300 – 320°C
This when tested about 1/2 Ferric.

Found reference Z fur angewandt Chemie 1893 page 201
D.R.P 67978 Molten iron containing .1 to 2 per cent manganese add per every 10 tons (Chinese tons) 100 Kilos FeS_2 in a way that the FeS makes thorough contact with all the iron the Manganese Sulphide is immediately formed and floats without melting on top of the iron
an Iron of 1.9 % Manganese was reduced in this way to .1 % Mn.

Copy Mr. Edisons Note

Boil strong sol of $FeSO_4$ with some $MnSO_4$ add a little NaOH to make a small precipitate keep boiling for 1/2 hour cover the dish and have some beads in to keep all Ferrous Then filter & Test. precipitate [illegible]

Tried according to Note Filtered off all was green practically none had gone to ferric. Took Filtrate and precip Iron as basic acetate filtered, Filtrate added Bromine Much Manganese Manganese is not removed by this method so-called Ferric Iron, no bottle

170 °C = 338 °F

Oxidized $FeSO_4$ with Nitric Acid heated to 170 °C for. 8 hours then cooled and put in bottle.

Took some added water, boiled, all dissolved another portion added cold water 9.00 AM 12 noon 1/2 was dissolved 3 PM 3/4 dissolved and at somewhere between 4 - 5 PM all dissolved this does not agree with Olsen who says $Fe_2(SO_4)_3$ slightly soluble in cold water and decomposes in hot water.

Some left out in Pan at 5.00 PM would see spots where it deliquesced (became liquid).

Tried Pure $Fe_2(SO_4)_3$ made from Silver Lake $FeSO_4$ Oxidized with HNO_3 and Heated until no more SO_3 came off but not so high as to form Fe_2O_3 this dissolved practically at the same rate

5 grams of $Fe_2(SO_4)_3$ + Manganese Sulphate 50 cc water, room temp, stirred 5 min Filtered rapidly thru Vacuum Filter washed 100 cc H_2O (room temp). Quite a little Manganese left in the Iron and too much iron in the wash water.

Effect of Various Chemicals on Ferrous & Ferric Sulphate and Mixtures

Name of Chemical	$FeSO_4$	$Fe_2(SO_4)_3$	$MnSO_4$	$FeSO_4$ $MnSO_4$	$Fe_2(SO_4)_3$ $MnSO_4$
Ba peroxide N.S.	cold or hot no ppt if enough added since Fe carries down acid only $BaSO_4$?	white ppt. cold or hot $BaSO_4$?	cold or hot Brown precip. $Fe_2(SO_4)_3$ and $BaSO_4$	Pale brown ppt. hot or cold and in and of slightly acid	Brown ppt. cold but hot Fe ppt
H_2O_2 N.S.	No precip	No precip	No ppt	No precip	No precip.
Ammon persulphate	No precip cold or hot	No precip cold or hot.	More cold MnO_2 Hot.	No precip. cold but all made Ferric	ppt. MnO_2 out when hot.
Na perchlorate N.S.	No precip cold or hot	No precip cold or hot	No precip cold or hot.	No ppt	No ppt
Na perchlorate N.S.	precip. cold. more if enough Fe precipitated when it boiled	pale red hot but not if enough Free Acid	precip. cold or hot but not if enough Free acid	Precip	Precip
Ba Sulphide	cold precip hot precip in acid sol.		precip in cold also in acid sol. $BaSO_4$?		

Barium peroxide precipitates manganese from Manganese Sulphate both Hot or cold in of [illegible] and no a nearly black precipitate

Ferrous Sulphate cold or hot a brown precipitate of sol. a little acid only a ppt of $BaSO_4$ (white)

Ferrous Sulphate + Manganese Sulphate (mixture) Black precip of manganese

Ferric Sulphate + Manganese brown ppt of Manganese

Ferric Sulphate no manganese brown ppt

Barium Peroxide gives a precipitate in both Ferrous & Ferric Solutions but this consists of a little Manganese Iron and Barium Sulphate if acid nearly all Barium Sulphate. Manganese is still found in the filtrates

Sodium Peroxide

Exp # 1153

Experiment on Dimethylglyoxime & α Benzildioxime for Nickel.

8.0 grams Ferrous Sulphate
1.5 grams Copper Sulphate
.5 Nickel Sulphate
Made up to 100 cc Took 10 cc for analysis

1 Precipitating with dimethyl — direct after oxidizing!

Total = 14.1550
Filter = 14.1054
.0496 = .0100 g Ni

Nickel Dox —

= 0.100 g Ni in 100 cc

2 Precipitating with α Benzil direct after oxidizing

Total = 14.1541
Filter = 14.0920
.0621 = .0067 g Ni

Ni Benzil —

= .067 g Ni

3 Precipitating Cu out first with H_2S and Sol. then boiling off H_2S oxidizing with H_2O_2 then made slightly ammoniacal precip with Dimethyl — after adding Tartaric Acid —

Total 14.0900
Filter 14.0392
.0508 = .0103 g Ni

Ni Dimethyl

= 0.103 grams Ni per 100 cc

4 Same as # 3 except precip Ni in Ammoniacal Sol with Benzil —

Total = 14.1806
Filter = 14.0830
.0976 = .0106 g Ni

Ni Benzil

= .0106 grams Ni per 100 cc

5 Cu left in Fe precipitated out with NH_4OH after oxidizing, then Ni precip out of the ammoniacal Sol with Dimethyl

Total = 14.1385
Filter = 14.1115
.0270 = .00548 grams Ni

Ni Dimethyl

= 0.0548 g Ni in 100 cc

6 Same as # 5 except precip with Benzil
Cu in Fe precip out NH_4OH

Total = 14.1410
Filter = 14.0830
.0580 = .00634 grams Ni

= 0.0634 g Ni per 100 cc

50 grams Fe_2O_3 taken from Silver Lake put into a percolator covered with concentrated C.P. Hydrochloric Acid left stand over Night then opened pinch cock at bottom and drained washed with water in percolator then washed well in vacuum filter hot water dried weighed, weight 25 grams 50% had been dissolved.

50 grams Fe_2O_3 treated same as above except used 50% H_2SO_4 Weight after drying 45 grams. loss 10%.

225 grams of Iron Oxide Mix # 6689 Put into a percolator added 500 cc 5% H_2SO_4 let stand 24 hours then opened pinch cock on bottom drained which took 6 hours then closed pinch cock added 500 cc Water let stand 24 hours then drained 6 hours added another 500 cc Water let stand 24 hours then drained 6 hours. took Sample and dried without further washing Kept over hot plate at 30-35°C during entire operation.

Found .0093% Manganese based on the equivalent of Metallic Iron.

Original Sample from Silver Lake contains 0.017 Manganese based on the Metallic Iron

1 gram Fe_2O_3 required .4 cc $\frac{1}{10}$ N Fe Ammon SO_4

Value Fe Sol in Mn for 1 cc

$= .4 \times .001632 = \frac{.00065228}{.699} = .00093$

$= .0093\%$ Mn on the Fe Basis

13.8717
13.8680
.0037 loss in melting.

Crucible 13.8680
12.5320
1.3360

13.8717 Total
12.5320 Crucible
1.3397
1.3360 check.
.0037 Loss in melting

No trace Chloride

1.3360 to 200 cc Tools 10 cc stock for determination of Sulphates & Chlorid also K direct

Glass 13.5500 Total 14.1920
14.0380
.1540 K Plat Chloride

x .35846 = .0552 K_2SO_4 x 20 = 1.1040

Total 12.6229
Crucible 12.5320
.0909 x .343 = .0311787 SO_3
$BaSO_4$
x 2.1765 x 20 = K_2SO_4 1.35716

1.3360 Sulphates
1.1040 K_2SO_4
.2320 Na_2SO_4
converted to Na_2SO_4

.2320 / 1.3360 = 17.3 %

$K_2SO_4 + 2NaCl = Na_2SO_4 + 2KCl$

1 st Treatment

25 cc Taken for Fe Total 500 cc

Total 12.7960

Crucible 12.5320

.2640 Fe X20 = 5.2800 grams Fe_2O_3

from 225 = 2.34 % Loss

Total 12.8040

12.5320

.2720 X20 5.7400 grams Fe_2O_3

= 2.55 %

2.34 %

4.89 % Total loss of Fe_2O_3

1 g. required .3 cc

.3 X .001632 $\frac{.0004896}{.669}$ = .00073

= .0073 % Mn based on the Fe Content

Original Mn based on the Fe content = .017 %

First Treatment Mn " " " " " = .0093

Second " " " " " " " = .0073

5.8090 3.5973

8.7135 5.846

17.427 11.692

174.27 58.46

K_2SO_4 + 2 NaCl = Na_2SO_4 + 2 KCl

Crucible 12.5330

Total = 13.1365 after Heating 13.0940

Crucible = 7.5170 7.5170

5.6195 actual Weight 5.5770

5.5770

.0425 Brought up to 200 cc

1 Chlorides Total = 23.1120 50 cc

1 Sulphates Crucible = 21.1190

1 K. $BaSO_4$ 1.9930 in 50 cc

1.9930 $BaSO_4$ X .343 = .6835990 SO_3 X 2.1765

= 1.48785 K_2SO_4 X 4 = 5.9514 Total K_2SO_4 in

10 cc

Total = 14.7010

Total Gloss = 14.1510

K_2PtCl_6 .5500 X .35846 = K_2SO_4

X 20 = 3.89906 K_2SO_4

.6835990 X 4 = 2.73439 X 2.1778

= 5.954967 K_2SO_4 4.4149 X .3779

= 5.9549 = 1.6683 Na_2SO_4

5.5770 3.9082 K_2SO_4

.3779 5.5770 Total Sulphates

Exp. # 1154

Finely Divided Nickel

1

April 30/20

Exp # 1155 Porous Nickel Hydrate

262.85 80.02

$NiSO_4 6H_2O + 2NaOH = Ni(OH)_2 + Na_2SO_4$

131.4 42

NaOH Titration 13.8806
13.5500
.3306 g NaOH to 250 cc took 10 cc

1st 7.95 cc H_2SO_4 .005053
2nd 8.00 cc
7.98 cc average

= 99.4% NaOH

#1 1314 grams $NiSO_4 6H_2O$ ground fine
42 " NaOH " Regular P. Bat
Mixed with spatula No Water
passed through rolls 10 times
between 6 – 7 times seemed sufficient
but went to 10
Dried 1/2" overhot plate
Keep Ground $NiSO_4 6H_2O$ in air tight jar
" " NaOH " " " "
while not in use.
First pass through rolls space about 1/16
gradually tightened up after each pass.
First pass reaction commenced pitty moist
all over and steaming a little.

2

131.4 grams $NiSO_4 6H_2O$ ground fine
2% Excess NaOH 42.84 " NaOH " Reg. P. Bat.
Mixed with Spatula NO WATER
passed through rolls 10 times
Washed by percolation 5 times
Dried on hot plate over night

3

131.4 grams $NiSO_4 6H_2O$ ground fine
4% Excess NaOH 43.68 " NaOH "
Mixed with Spatula NO WATER
passed through rolls 10 times
Washed by percolation 5 times
Dried on hot plate over night

20 Mesh Glass rolled in Ni Oxide with just enough moisture to make it stick. Dried and reduced in H. Ni reduced but did not adhere to the glass.

Ni Chloride dried in shallow pan until scaly then peeled off with spatula but in H reduced gave a metal flake but the particles did not adhere. Went to fine powder when rolled. so heated to many fusion particles stuck together a little. Note the dry scaly chloride takes up moisture slowly.

Ni Carbonate dried and also heated to Oxide were reduced in H. at a heat about 900°F (guessed) the particles are very lumpy and hard powder, easily at a lower temp the particles remain as a fine powder (black black) do not condense very well when loose, but good when pressed together

Reduced $Ni(OH)_2$. Ni OH heated to Oxide both reg. mesh as used in loading machines and 60-on 80

all reduced H. red heat then pressed under hydraulic 500 – 1000 – 2000 lbs on 5" ram. makes flakes between 1000 – 2000 also tried to roll out flakes between rollers. both methods have the fault that the thickness can not be controlled so that fines and thick flake all come together.

Tried to coat pieces of carbon with Ni by soaking in $Ni(NO_3)$ dying soaking again and reducing coated all over with metallic Ni but all in spots that is it shows shrinkage cracks.

~~Tried~~

A Reduced $NiCO_3$ direct in iron retort bringing delivery tube to bottom of retort and covering with a screen. Passed H breaks let H run until cool.

Schedule

245	thermometer could not be read
255	
305	
315	392°F
325	464°F

		°F
335		536
345		590
355		644
405		672
415		824
425		968
435		968
445		932
505	1 hour 20 min	957
515		950
525		957
535		950
545		950

Lumps found on edges where material touched retort

Repeated same experiment as # A except $NiCO_3$ was put in a porous cup within the Iron retort to prevent material from touching the sides

over

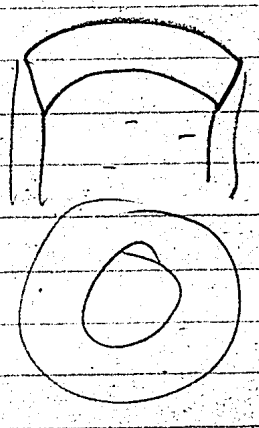
Time Schedule		°F
10.00		
10.10		
10.30		356
10.45		626
10.50		770
11.00		968
11.15		932
11.30		924
11.40	1 hour 15 min	968
11.50		960
12.00		950
12.10		968
12.15		968

3.89906
1.6683

K_2SO_4 Indirect 3.9087
" " by Platinic Chloride 3.89906
.019

3 1/2 %

3 % to 4 1/2 % Hg O.



47
10 Sprd 2500cc same as 40 mg
20 Lime
1g Glycerine

45 : 10g Ammonium
10 Lime
2 1/2 Magnesium chloride

44 250)2000(
10 g ammonite
10 g Lime 30 8 .24 .024
2 1/2 g NaCl .024 .024
6

33 10 g Sprd
10 g Lime

32 20 g Sprd 294
10 g Lime 2
5.8
6.2

1 cc of $MnSO_4$ sol = .020182 $MnSO_4$

1 cc = 0.00734 Mn.

Pt Cruc. 12.5434 6

Amblygonite

Al_2O_3	36.88	34.5
Lithia	6.68	6.9
Na	3.29	6.0
K	.43	
HPO_4	47.58	47.9
Fluorine	8.11	8.4

Spodumen

Silica	65.02
Al_2O_3	29.04
FeO	1.42
Lime	.97
Magnesia	.07
Lithium	4.49
Soda	.07
K	.07

Lepidolith

SiO_2	50.32	46.52
Al_2O_3	28.54	21.81
Fe_2O_3	0.73	4.68
Lime	1.01	—
Magnesia	.51	0.44
Rubidium	.24	
Cæsium	trace	
~~Li~~ Fl	.99	
Na Fl	1.27	
K Fl	12.06	
FeO		6.80
MnO		1.96
MnO_2		

K	—	9.09
Na	—	.34
Li	—	1.27 — 5.67
Fluorine	—	7.47
Water	3.12	
HPO_4	—	0.13